



Research Article

INVESTIGATION OF THE EFFECTS OF BORIC ACID AND BORIC ACID WASTE ON THE ALKALINE ACTIVATION OF BLAST FURNACE SLAG

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Received: 12.01.2018 Accepted: 15.02.2018

ABSTRACT

The cement industry generates approximately 5% of man-made carbon dioxide (CO₂) emissions worldwide. Today, research interest in reducing carbon dioxide emissions in the cement industry is increasing rapidly. For this purpose, alkaline-activated materials or geopolymers, have become an important research topic. Alkaline-activated materials use waste such as fly ash, blast furnace slag, and silica fume without any clinker. Alkaline-activated binders have been investigated for many years owing to their environmentally friendly properties, but they have not progressed beyond research activities owing to production problems and a lack of standardization.

In this study, the effects of retarders on mortar samples produced by alkaline activation were investigated. Blast furnace slag obtained from Eregli was used as the binder. Alkaline activation was carried out using a mixture of sodium silicate and sodium hydroxide as the activator. The setting times were delayed using boric acid and boric acid slurry obtained from Etimaden Bandırma plants. The effects of boric acid and boric acid waste on compressive strength, setting time, and consistency were determined. The carbonation reactions, chloride migration coefficients, alkaline silica reactions, and water absorption properties of the mortar mixtures were determined according to EN 13295, NT Build 492 standard, the accelerated mortar method and ASTM C1403 standard, respectively.

Keywords: Boric acid, boric acid waste, alkaline activation, blast furnace slag.

1. INTRODUCTION

The cement industry produced 1.45±0.20 Gt/year of carbon dioxide (CO₂) emissions worldwide in 2016. Approximately 8% of total man-made CO₂ emissions come from cement production [1]. According to Turkish Cement Manufacturer's Association statistics, in Turkey, approximately 75 million tons of cement was produced in 2016 [2].

The use of fossil fuels, deforestation, and the decomposition of carbonates are the most important contributors to human-driven CO₂ emissions around the world [1]. CO₂ emissions generated from the burning of fossil fuels to produce the heat required for cement manufacture can be reduced by using alternative fuels and energy efficiency methods. However, the formation

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of CO₂ resulting from the basic decarbonization reaction in cement production cannot be prevented [3].



Therefore, it is important to develop and use alternative binders with low carbon content, rather than cement, for environmentally friendly construction. In this regard, alkaline activation methods of mortar and concrete production are promising. The method is based on activating materials containing calcium and aluminum silicate, such as fly ash, blast furnace slag, silica fume, and metakaolin, using some alkalis, such as sodium hydroxide and sodium silicate. Aggregate serves as the skeleton in the building, as it does in the production of traditional concrete.

Activation with alkalis was first described and then patented by Kühl in Germany in 1908 in the form of slag cement. Kühl reported that the reaction of an alkaline source with a precursor containing alumina and silica gives a hardening product that is comparable to Portland cement [4]. The topic was studied by Purdon and other researchers in 1950 and they reported that the compressive strength enhancements of the resulting products were similar to those of Portland cement, and the products were even superior to Portland cementitious structures in terms of generated heat, bending strength, and solubility [5, 6].

Products activated with alkalis have been defined by various researchers as mineral polymers, inorganic polymers, inorganic polymer glasses, ceramics bonded with alkalis, alkaline-ash material, soil cement, soil silicates, KJ-connector, F-concrete, hydroceramic, zeocement, and zeoceramic. Moreover, Prof. Dr. Davidovits named the products obtained by the activation of low-calcium alumina silicates, patented in 1979, as geopolymers [8]. Geopolymers have been reported to exhibit pseudo-zeolitic structures instead of chain calcium silicate structures [10, 11]. In many cases, it has been reported that geopolymers are a subgroup of alkaline-activated materials [7, 9].

Research on activation with alkalis has increased rapidly, especially since the 1990s. Different problems of alkaline-activated materials have been highlighted by various researchers in relation to Portland cementitious materials. The use of caustic materials at high concentrations, sensitivity to added water, efflorescence, and setting problems are some of the main problems with the alkaline activation process [13-15]. Nevertheless, it has been reported that the durability of the products is better than that of Portland cement structures, such as in terms of resistance to sulfates, acids, and alkaline aggregate reaction [16-20]. In some studies, alkaline-activated materials have been reported to be effective at stabilizing radioactive materials and hazardous waste [21, 22].

Several patents have reported that boric acid has a retarding effect on the alkaline activation process [23-25]. However, the effects of boric acid and boric acid waste on the alkali activation process have not been studied in detail.

In this study, the effects of boric acid and boric acid waste on the alkaline activation of blast furnace slag were investigated.

2. MATERIALS

Blast furnace slag obtained from the Erdemir (Zonguldak, Turkey) was used as the binder. CEM I standard sand supplied from Limak Cement Co. (Ankara, Turkey) was used as the aggregate. Analytical-grade sodium hydroxide and sodium silicate obtained from AS Kimya (Istanbul, Turkey) were used as the alkaline activator. Boric acid (analytical purity) and boric acid waste (slurry) obtained from Eti Maden Bandirma Boron and Acid Plants (Balıkesir, Turkey) were used as retarders. The contents of the blast furnace slag and boric acid waste are detailed in Table 1 while the properties of the alkaline activators are reported in Table 2.

Table 1. Chemical compositions

	CaO (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	MgO (%)	K ₂ O (%)	Cl (%)	Na ₂ O (%)	SO ₃ (%)	B ₂ O ₃ (%)
BFS	25.98	41.58	7.13	1.28	5.91	0.57	0.024	0.62	1.33	-
Slurry	16.41	21.31	1.24	0.35	19.41	0.68	-	6.47	-	11.6

Table 2. Properties of alkaline activators

Activator	Molarity (M)	Density (g/cm ³)	Solid Content (%)	Ms Module (SiO ₂ /Na ₂ O)
Sodium silicate	-	1.558	53	4
Sodium hydroxide	12	1.34	36	-

3. EXPERIMENTAL STUDIES

3.1. Preparation of Mixtures and Determination of Properties

Table 3. Compositions of mortar mixture samples

		Aggregate	B.	S.S.	S.H.	W	Adm.	Activator Solids	Total Water	W/B
Y4	g	1350,00	450,00	192,89	77,11	48,60	7,20	142,72	175,88	0,39
Y5	g	1350,00	450,00	225,04	89,96	21,60	7,20	166,51	170,09	0,38
Y6	g	1350,00	450,00	225,04	89,96	14,40	10,80	166,51	162,89	0,36
Y7	g	1350,00	450,00	225,04	89,96	10,80	21,60	166,51	159,29	0,35
Y8	g	1350,00	450,00	225,00	89,96	28,80	21,60	166,49	177,28	0,39
Y9	g	1350,00	450,00	192,17	89,96	36,00	21,60	147,44	170,69	0,38
Y10	g	1350,00	450,00	266,74	106,70	0,00	21,60	197,39	176,05	0,39
Y10-1	g	1350,00	450,00	266,74	106,70	0,00	64,80	197,39	176,05	0,39
Y11	g	1350,00	450,00	266,74	106,70	0,00	10,80	197,39	176,05	0,39
Y11-1	g	1350,00	450,00	266,74	106,70	0,00	32,40	197,39	176,05	0,39
Y12	g	1350,00	450,00	339,61	135,85	0,00	21,60	251,31	224,15	0,50
Y13	g	1350,00	450,00	213,39	106,70	23,76	16,20	166,45	177,40	0,39
Y14	g	1350,00	450,00	169,85	169,85	4,14	16,20	166,45	177,39	0,39
Y15	g	1350,00	450,00	0,00	295,63	0,00	16,20	118,25	177,38	0,39
Y16	g	1350,00	450,00	266,74	106,70	0,00	18,02	197,39	176,05	0,39
Y16-1	g	1350,00	450,00	266,74	106,70	0,00	54,06	197,39	176,05	0,39
Y17	g	1350,00	450,00	225,00	89,96	28,80	18,02	166,49	177,28	0,39
Y18	g	1350,00	450,00	88,20	35,28	117,36	7,20	65,27	175,57	0,39
Y19	g	1350,00	450,00	266,74	106,70	0,00	0,00	197,39	176,05	0,39
Y20	g	1350,00	450,00	266,74	106,70	0,00	5,00	197,39	176,05	0,39
Y20-1	g	1350,00	450,00	266,74	106,70	0,00	15,00	197,39	176,05	0,39
Y21	g	1350,00	450,00	266,74	106,70	0,00	27,00	197,39	176,05	0,39
Y21-1	g	1350,00	450,00	266,74	106,70	0,00	81,00	197,39	176,05	0,39

Mortar mixtures were prepared according to the modification specified in European standard EN 196-1. Solid and liquid materials were blended separately and then homogenized. The solid material was slowly added to the liquid mixture placed in the mortar mixer at a low mixer speed over the course of 1 minute and then the mixer speed was increased to a higher rpm. The samples were kept for 90 seconds and then mixed at high speed for 60 seconds. Samples of 4 × 4 × 16 cm were used for bending and compressive tests and a vibratory table was used where placement was

required [26]. The compositions of the mortar mixture samples prepared for the experimental studies are detailed in Table 3.

Setting time, consistency, and compressive strength tests were carried out according to TS EN 480-2, TS EN 413-2, and TS EN 196-1, respectively. The prepared mixtures were also subjected to alkaline silica, chloride impermeability, and carbonation tests for durability [26-28].

3.2. Durability Properties

Alkaline Silica Reaction Test: The soluble reactive silica in the aggregate reacts with the alkalis in the cement, water, and additive, causing elongation and cracking in the mortar. This reaction is determined by measuring length extensions in mortar bars.

The alkaline silica test was carried out using the accelerated mortar method [29]. For this purpose, length extensions were measured at 2, 7, and 14 days after keeping the samples in a 1M NaOH solution at 80°C.

According to the results of a test performed using the reference CEM I 42.5A type cement, a natural sand sample exhibiting the alkaline silica hazard was used.

Chloride Impermeability Test: Nowadays, concrete frequently is used with steel reinforcements. Chloride ions—originating from harmful environments under the influence of concrete—damage these steel reinforcements. This is especially important for concrete structures that are affected by sea water, such as bridges. The steel reinforcements used in concrete are subject to corrosion over time, which substantially reduces the service life of the structure. One of the major causes of the corrosion of concrete is the passage of chloride ions through the concrete [30]. The chloride impermeability test was applied to the cast mortar samples prepared in the laboratory environment. The chloride migration coefficient is determined by measuring the resistance of the tested material against the penetration of chloride ions. The higher the resistance, the lower the migration coefficient. The test setup was established in the laboratory according to the NT Build 492 standard (Figure 1) [31].

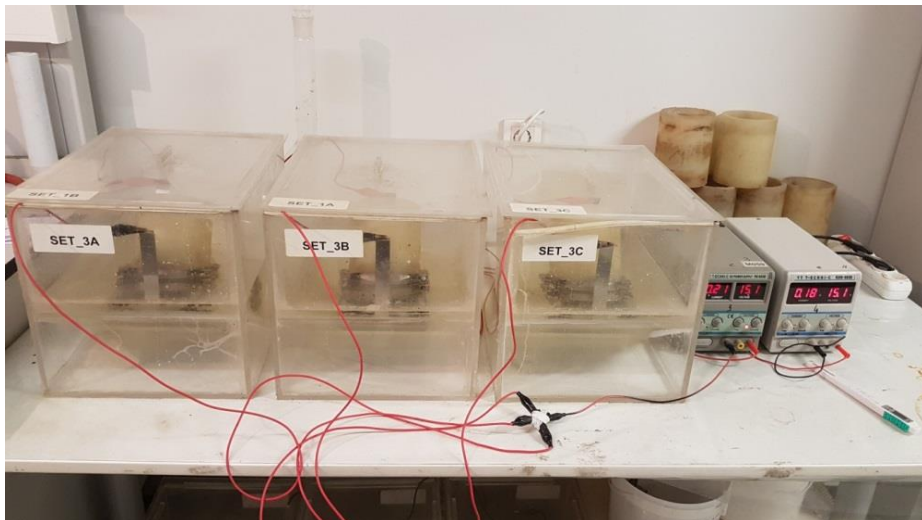


Figure 1. Chloride impermeability test setup

Carbonation Experiment: Cement hydration products, especially calcium hydroxide (CaOH), react with CO₂ in the air and rainwater over time to form calcium carbonate (CaCO₃), a neutral

salt. Although the alkaline structure of concrete can naturally prevent corrosion the salts resulting from carbonation cause corrosion of reinforcements [32].

For the accelerated carbonation experiment, the samples were left in a concentrated CO₂ gas atmosphere for 56 days and the degree of carbonation was determined by measuring the carbonation depth of the concrete samples.

An atmospheric cabin with 1% CO₂ gas at 21°C and 60% relative humidity was used to store the samples. The CO₂ content, temperature, and relative humidity of the atmospheric cabin were measured and controlled automatically by the control units. The experimental setup was installed in the laboratory according to European standard EN 13295 (Figure 2) [33].



Figure 2. Carbonation experimental setup

4. RESULTS AND DISCUSSION

4.1. Setting Time and Consistency

The setting times and consistencies of the prepared mortar mixtures are reported in Table 4. The addition of boric acid and boric acid waste increased the setting times in all the mixtures. The effects of the amount of boric acid on the setting time are shown in Figure 3. It can be seen that boric acid waste (slurry) had a retarding effect of about 25–30% of the equivalent amount of boric acid.

It was observed that increasing the amount of boric acid resulted in a small increase (about 2–6%) in the mixture consistency. Boric acid was found to have retarding and plasticizer effects. When using boric acid waste, the plasticizer effect was not realized owing to the presence of substances such as lime in the slurry.

Table 4. Consistencies and setting times of mortar mixtures

Mix Code	Consistency	Setting Time (min)
Y4	16	10
Y5	16	10
Y6	15,5	20
Y7	15	300
Y8	17	660
Y9	16	360
Y10	17,5	360
Y10-1	17,5	420
Y11	17	15
Y11-1	17	20
Y12	Self compacting	75
Y13	16 – 17	17
Y14	16	7
Y15	<13	4
Y16	17	150
Y16-1	17	180
Y17	17	360
Y19	17	4
Y20	17	7
Y20-1	17	8
Y21	18	>1440
Y21-1	18	>1440

The use of boric acid was found to have a small delaying effect on the setting time up to a turning point, beyond which the addition of more boric acid was found to considerably affect the setting time. It was seen that the use of more boric acid than required may lead to the production of unsettled mortars. When only the amount of boric acid was changed, the setting time changes were constant up to 0.8% boric acid amount while a sudden change in setting time was seen between 0.8 and 1% boric acid content (Figure 3).

The amount of boric acid needed to obtain the required setting time was found to be dependent on the amount of alkaline activator, amount of binder, and ambient conditions, such as temperature.

Moreover, long-term observations made on mortars showed that the use of boric acid and boric acid waste improved the efflorescence effects resulting from alkaline activation.

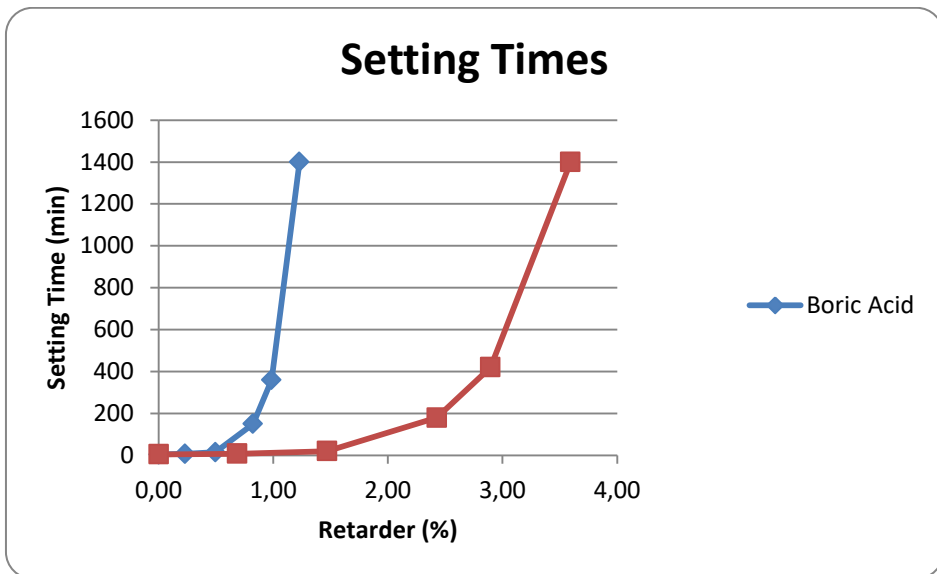


Figure 3. The effects of boric acid and boric acid waste on setting times

4.2. Compressive Strength

The prepared mixtures were kept for 1 hour at ambient conditions and then cured at 60°C for 23 hours in an oven. After curing, the samples were placed in water at 20±1°C and then used for the compressive strength test. The compressive strengths of the samples were measured at 1 day and 28 days.

Boric acid and boric acid waste (slurry) were added to mixtures with the same water/binder ratios, keeping the amounts of binder, activator, and aggregate constant. The effects of boric acid and boric acid waste (slurry) on the compressive strength are shown in Figure 4.

As the amount of boric acid was increased, the early 1-day strength decreased but the final 28-day strength increased. It was found that there was no significant increase in the compressive strength up to the optimum value of the amount of boric acid (turning point). However, 0.82% boric acid decreased the 1-day strength by 9.6% compared to the mortar mixture without boric acid and increased the 28-day strength by 8.9%. When using 0.98% boric acid, the compressive strength decreased by 16% in 1-day early strength while the 28-day compressive strength was increased by 6.9% compared to the mortar mixture without boric acid. It was found that the use of the optimum amount of boric acid caused an increase of the 28-day compressive strength of about 7–9%. The results showed that the use of more boric acid than required causes a significant decrease in the compressive strength.

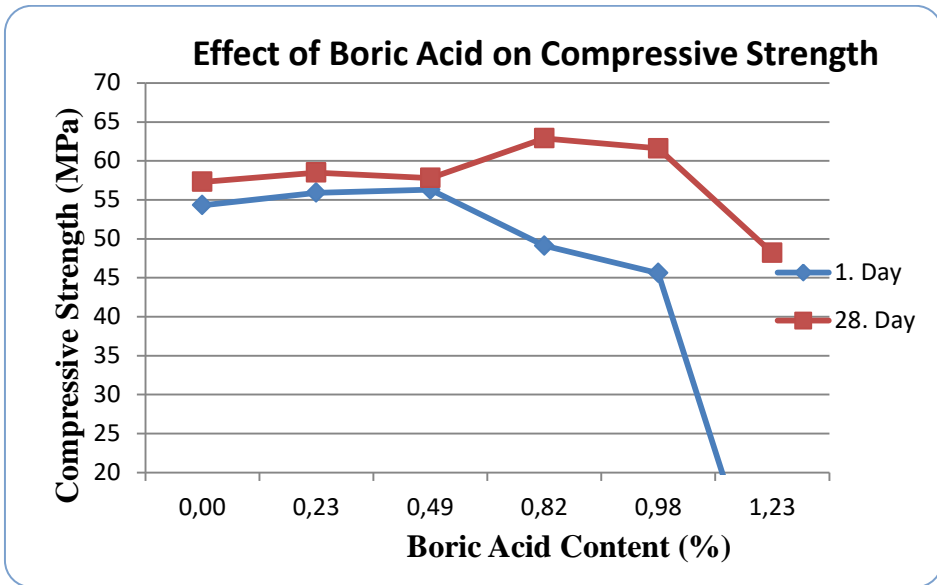


Figure 4. The effects of boric acid on compressive strength

4.3. Alkaline Silica Reaction

The alkaline-activated mortar sample showed 0.01% elongation on average at 3, 7, and 14 days. The equivalent natural sand sample showed 0.28% elongation at day 14 with CEM I 42.5R type cement. According to the results of the accelerated test method and experiments, it was found that the alkaline-activated blast furnace slag and boric acid mortar samples completely removed the aggregate hazard in terms of alkaline silica.

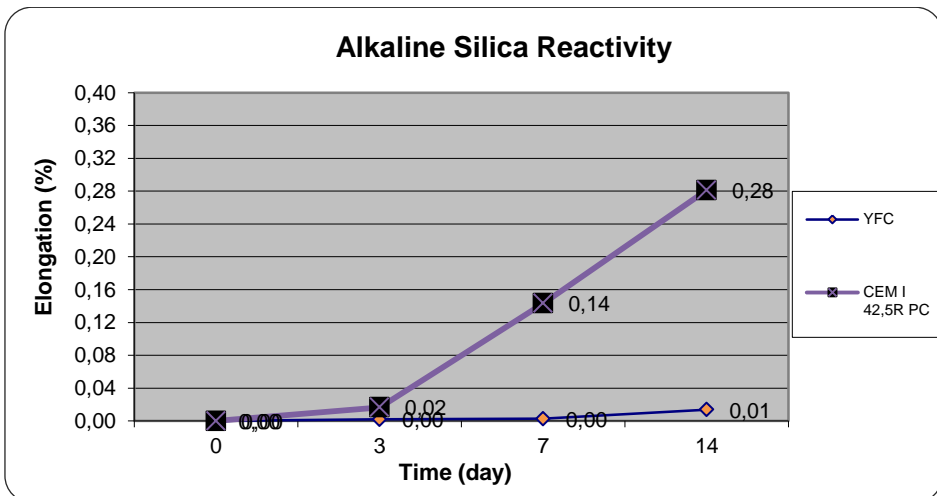


Figure 5. Alkaline silica reactivity experiment results

4.4. Chloride Impermeability

The effects of boric acid on the alkaline activation of blast furnace slag were determined by keeping the aggregate, binder, and water/binder contents constant. The reference test mixture was made using CEM I 42.5R type Portland cement. The compositions of the prepared mortar mixtures are detailed in Table 5.

Table 5. Mortar mixture compositions for chloride migration coefficient and carbonation experiments

		Aggregate	B	S.S.	S.H.	W	Adm.	Activator Solids	Total Water	Water/Binder
Y10	%	61,50	20,5	12,15	4,86	0,00	0,98	8,99	8,02	0,39
Y11	%	61,50	20,5	12,15	4,86	0,00	0,49	8,99	8,02	0,39
Y22	%	67,65	22,5	4,44	1,78	5,88	1,08	3,29	8,81	0,39
PC	%	66,7	22,2	-	-	11,1	-	-	11,1	0,5



Figure 6. Compositions of samples subjected to chloride impermeability test

It was determined that increasing the amount of boric acid in the sample mixtures (Y10 and Y11) with a high level of alkaline activator had a positive effect on the chloride impermeability test results and decreased the chloride migration coefficient by 35% for the prepared mixture compositions. When the amount of activator was decreased and the amount of water was increased instead of keeping the water/binder amount constant, the migration coefficient decreased to $1,56 \times 10^{-12} \text{ m}^2/\text{s}$ (Y10, Y11, and Y22).

No limit or category values are specified in the standards for the conformity evaluation of the chloride migration coefficient test results and the experimental results are limited by the application-oriented specifications. In the Turkey's 3rd bridge concrete conditions, the chloride migration coefficient limit value was determined as $3 \times 10^{-12} \text{ m}^2/\text{s}$.

According to TS EN 196-1, the chloride migration coefficient of a mortar sample prepared with Portland cement was found to be $17,8 \times 10^{-12} \text{ m}^2/\text{s}$. The chloride migration coefficients of the alkaline-activated blast furnace slag and boric acid mortar samples were significantly lower than those of the Portland cement mortars.

Table 6. Chloride migration experiment results

Measurements	Y10	Y11	Y22
Sample Thickness (mm)	47,43	49,07	48,02
Current Start (30 Volt) (mA)	120,1	120,1	19,9
Set Voltage (Volt)	15	15	50
Experiment Time (hour)	24	24	24
Current End (mA)	73,3	61,7	55
Depth of Penetration			
X_{d1} (mm)	4,75	8,51	6,4
X_{d2} (mm)	4,89	6,23	5,72
X_{d3} (mm)	5,35	6,3	8,71
X_{d4} (mm)	4,35	5,45	5,63
X_{d5} (mm)	4,27	6,85	6,1
X_{d6} (mm)	-	5,58	4,91
X_{d7} (mm)	3,91	5,64	6,49
$X_{Average}$ (mm)	4,59	6,37	6,28
Migration Coefficient ($\times 10^{-12} \text{ m}^2/\text{s}$)	3,15	4,87	1,56

4.5. Carbonation

The carbonation test results for the prepared mixtures are reported in Table 7. It can be clearly seen that all the samples were substantially carbonated. When mixtures Y10 and Y11 were examined, it was apparent that increasing the amount of boric acid negatively affected the carbonation, increasing the carbonation depth by about 8%. When the amount of alkaline activator was decreased and the amount of water was increased (Y11–Y22), the depth of carbonation increased by 12.6%. Comparing the test samples with the CEM I 42.5 R type Portland cement mortar mixture, the Portland cement showed better carbonation results than the alkaline-activated blast furnace slags. The reason for this may be that increasing the amount of boric acid has a negative effect on the carbonation by decreasing the mortar pH. A higher degree of carbonation was observed in the Y22 mixture because the carbonation experiment was dependent on the alkalinity of the waste.

Table 7. Carbonation experiment results

	Y10 Sample	Y11 Sample	Y22 Sample	Portland Cement Mortar
1. Measurement (mm)	14,44	13,41	14,76	3,25
2. Measurement (mm)	12,33	13,29	14,38	3,08
3. Measurement (mm)	13,12	12,10	14,80	-
Carbonation Depth (mm)	13,09	12,09	13,84	3,16
Carbonation Coefficient ($\text{mm}/\text{year}^{0,5}$)	33,2	30,6	35,1	8



Figure 7.Carbonation experiment mortar mixture compositions

Acknowledgement

This research has been supported by Yildiz Technical University Scientific Research Projects Coordination Department with Project number of 2013-07-01-DOP02.

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